DEN5406: Mass Transfer and Separations Processes I

Week 7: Filtration, Leaching (Extraction), Washing (& Dry Cleaning), .

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Separations and Syllabus Goals

Obtain Quantitative Understanding of the following processes:

Filtration - ✔ we started and continue! **We'll also cover this week: ✔**

Aggregation

✔ **Centrifugation**

Crystallization (controlled freezing) separation

Adsorption

Leaching (extracting metals from ores, making coffee, dry cleaning) ✔

✔ **Osmosis**

- **Forward Osmosis**
- **Reverse Osmosis**

Ion-exchange membranes

Drying

Distillation (controlled evaporation and condensation) ✔
and the many kinds of distillation

Applications: Surviving in Space, on a desert island without fresh water

What we will cover

By the end of this lecture you'll be able to:

Say what is **caking.** Other vocabulary: **washing, dewatering, feed conditioning, constant pressure filtration, Filter productivity, cycle time, Rotary vacuum drum filter, vertical disk filter, horizontal belt filter, Nutsche filter, Buchner funnel, plate-and-frame filter press**
gas filtration, impingement separators, overall on-flow area, wave-plate separators, deep bed filters, inertial and flow-line interception

Measure amount of caking and its effect on filtration
Quantify the relationship between caking and pressure drop
Different **types of filters** and their **mechanisms**

Devise strategies for mitigating **pressure loss** and prolong **filter life**

Quantitate the filtration capacity of single fibers

Efficiency and cost comparisons – Cost of material, and pressure drop
(pressure drop not only cost, e.g. max vacuum to generate by mouth in straws**)**

Recommended Reading

Available on Knovel – in the library:
De Haan & Bosch, Industrial Separation Processes, 2013, de Gruyter (Berlin)

Distillation Fundamentals and Principles, Gorak & Sorensen, eds., 2014, Elsevier

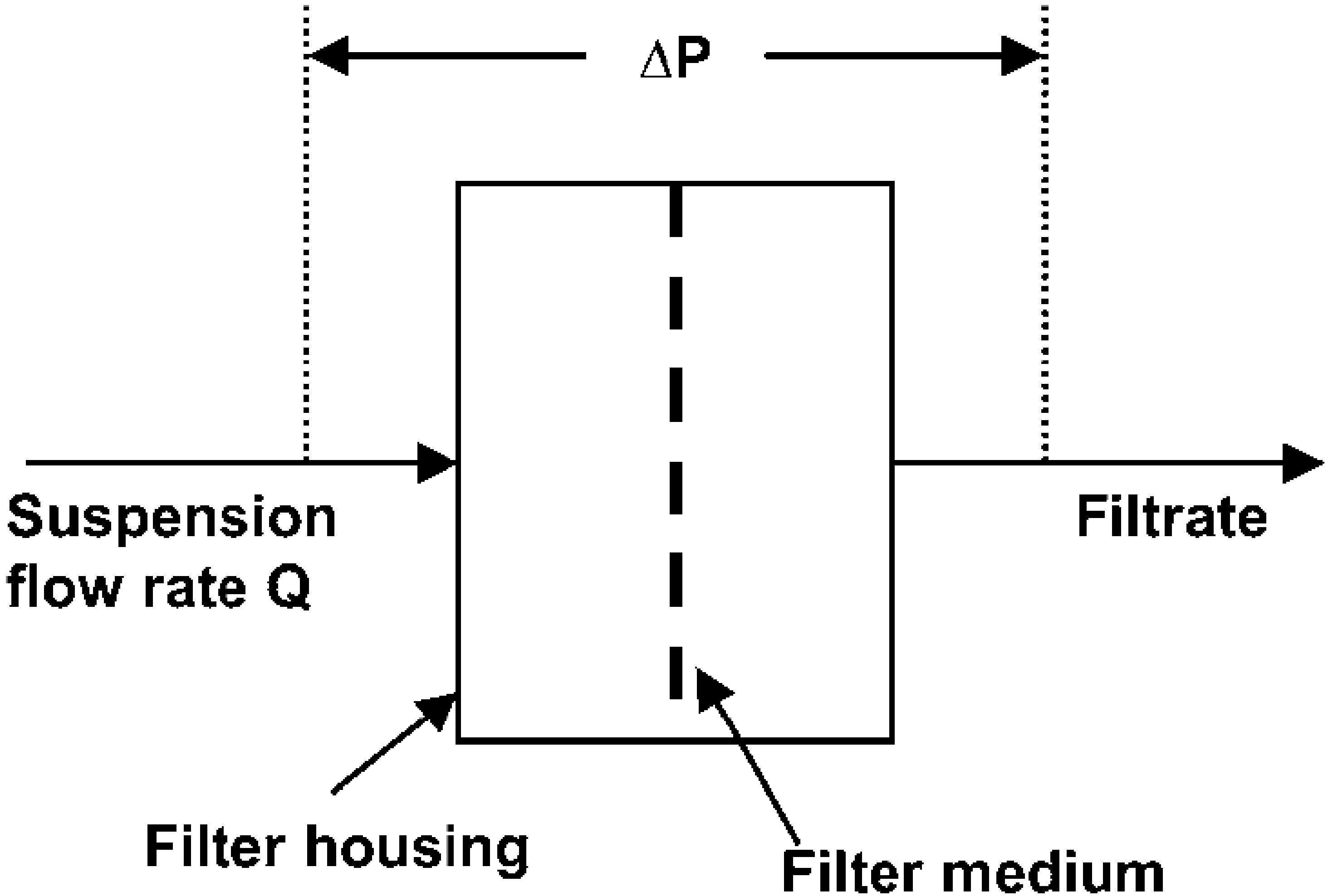
Reactive & Membrane-Assisted Separations, Lutze & Gorak, eds., 2016, de Gruyter

Also from
Seader, Henley, & Roper, Separations Process Principles, 2011, Wiley

Will assign pre-class reading -> will have a chance to discuss problems in class

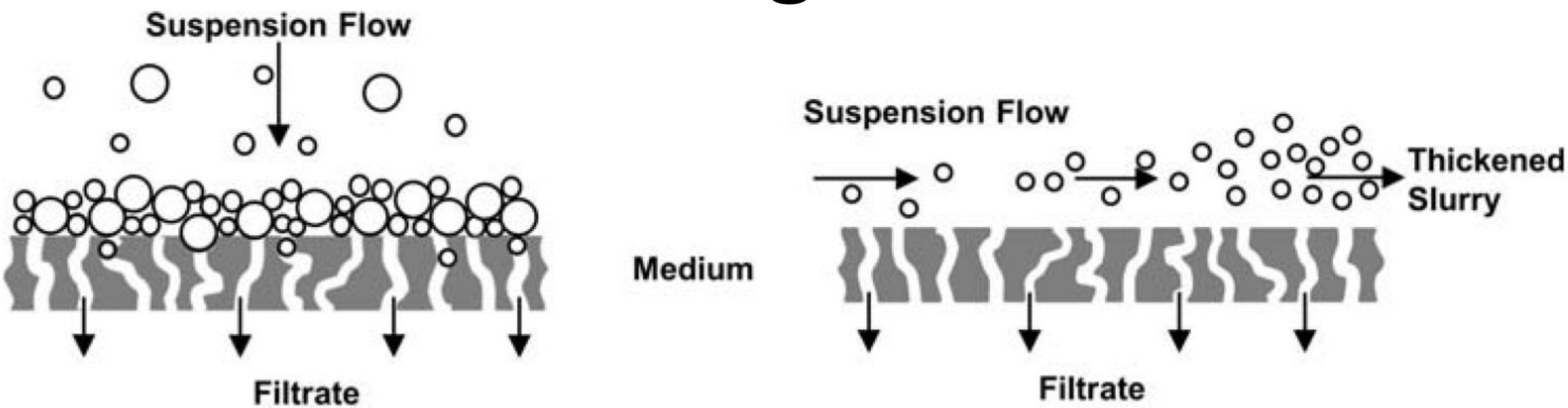
From Last Week:
Ch. 10 in
De Haan & Bosch, Industrial Separation Processes, 2013, de Gruyter (Berlin)

Filtration



Efficiency of filter = % particles retained (of a given size)
Efficiency vs. pressure drop (lower pressure is better)

Filtration – Caking and membranes



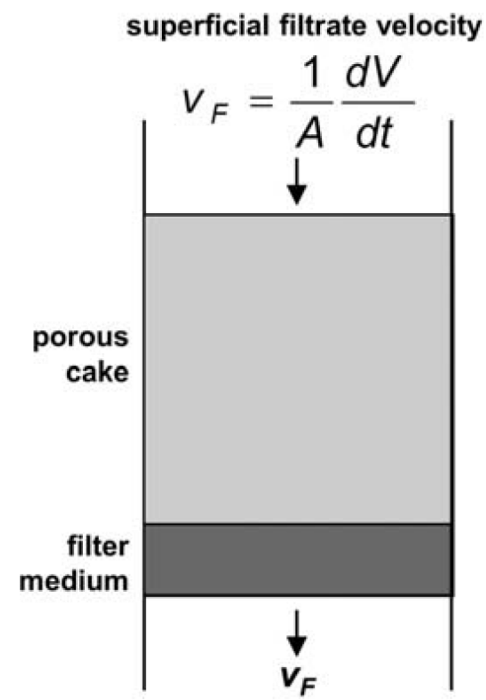
Cake vs. Cross-flow

Filter Porosity

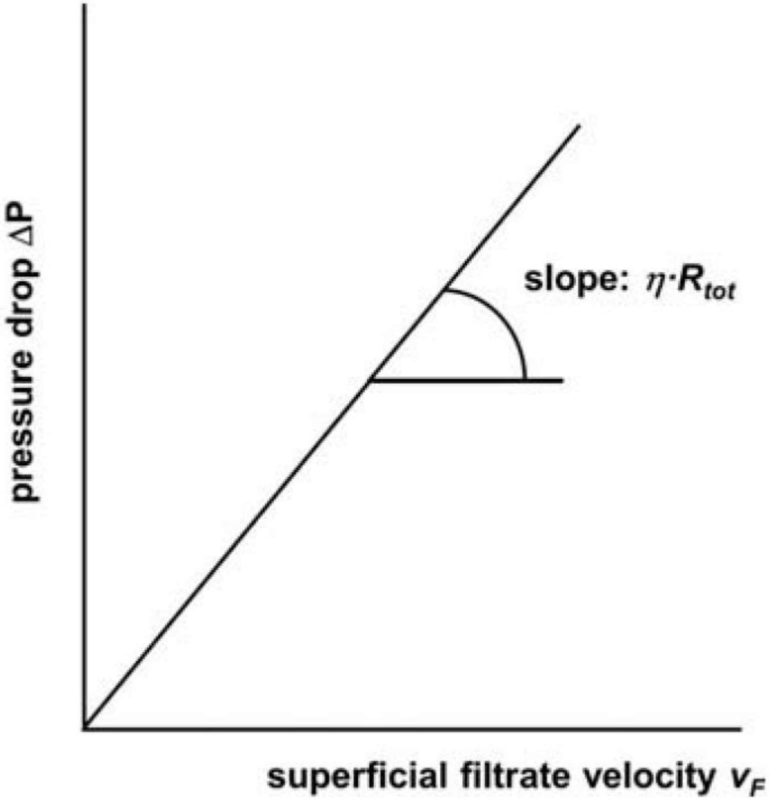
$$\varepsilon = \frac{volume\ of\ voids}{total\ bed\ volume}$$

Filtrate Velocity

$$v_F = \frac{\Delta P}{\eta\, R_{tot}}$$



Filtration – Quantitation



$$v_F = \frac{\Delta P}{\eta (R_M + R_C)}$$

Resistance due to filter medium
And to cake (R_M and R_C)

$$v_F = \frac{1}{A} \frac{dV}{dt}$$

$$\Delta P = \Delta P_C + \Delta P_M = \eta \frac{R_C + R_M}{A} \frac{dV}{dt}$$

Darcy's Law

De Haan & Bosch, Industrial Separation Processes, 2013, Ch 10.

Filtration – Problem Solving 1

Filtering cold water ($\eta = 10^{-3}$ Pa.s) always topping up to 10.2 cm water, yields 250 cm^3 in 1 min. The effective coffee filter area A is 0.0025 m^2. 1bar =10^5 Pa= 1020 cm column of water. How many Pa is the pressure?

$$v_F = \frac{1}{A}\frac{dV}{dt}$$

What is the resistance of the coffee filter paper?
Later you pour the same height of coffee, at 80 °C, ($\eta = 0.333 \times 10^{-3}$ Pa.s) and collect 200 ml in 1 min.
What is the resistance of the coffee grinds?

$$v_F = \frac{\Delta P}{\eta\,(R_M + R_C)}$$

Try to define the quantities for the equation yourself
It's not too hard – mostly identifying the words that
Go with the symbols and plugging in.

The goal of this is literally to change your brain.
Looking at the solution does a bad job of that.

Filtration – Problem Solving 1

Filtering cold water ($\eta = 10^{-3}$ Pa.s) always topping up to 10.2 cm water, yields 250 cm^3 in 1 min. The effective coffee filter area A is 0.0025 m^2. 1bar $=10^5$ Pa= 1020 cm column of water. How many Pa is the pressure?

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What is the resistance of the coffee grinds?

$$v_F = \frac{\Delta P}{\eta\,(R_M + R_C)}$$

Sometimes there are no solutions – need your own but here's a hint:

$$\Delta P = \Delta P_C + \Delta P_M = \eta\,\frac{R_C + R_M}{A}\frac{dV}{dt}$$

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$$v_F = \frac{1}{A}\frac{dV}{dt}$$

$$v_F = \frac{\Delta P}{\eta\,(R_M + R_C)}$$

Sometimes there are no solutions – need your own.

$$v_F = \frac{1}{A}\frac{dV}{dt} = \frac{250\ cm^3}{25\ cm^2\ min} = \frac{10\ cm}{min} = \frac{1\ cm}{6\ s} = \ldots \frac{10^3 Pa}{10^{-3} Pa.s * R_M}$$

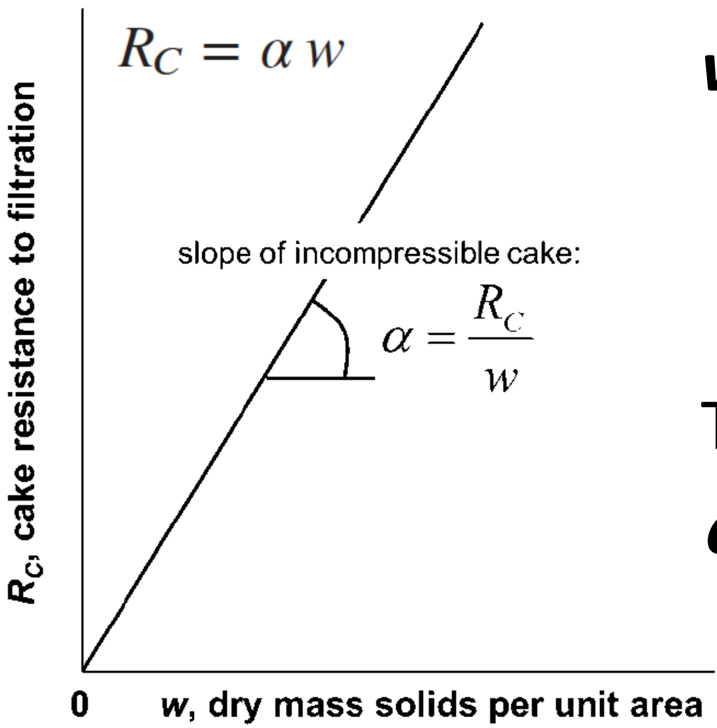
$R_M = 6 \times 10^6$ /cm , so when $v_{Fcoffee} = 0.1$ cm/s &

$\eta = 0.333 \times 10^{-3}$ Pa.s, $R_M + R_C = 30 \times 10^6$/cm, **$R_C = 27 \times 10^6$/cm**

Filtration – Incompressible Cake

<u>Incompressible cake</u> causes:

a *linear* increase in resistance $\boldsymbol{R_c}$ with *cake height*



w is <u>mass dry solids per filter area A</u>

$$w = \frac{c\,V}{A}$$

[kg/m2] where c is kg solids per volume V of suspension

The proportionality slope $\boldsymbol{R_c / w}$ is

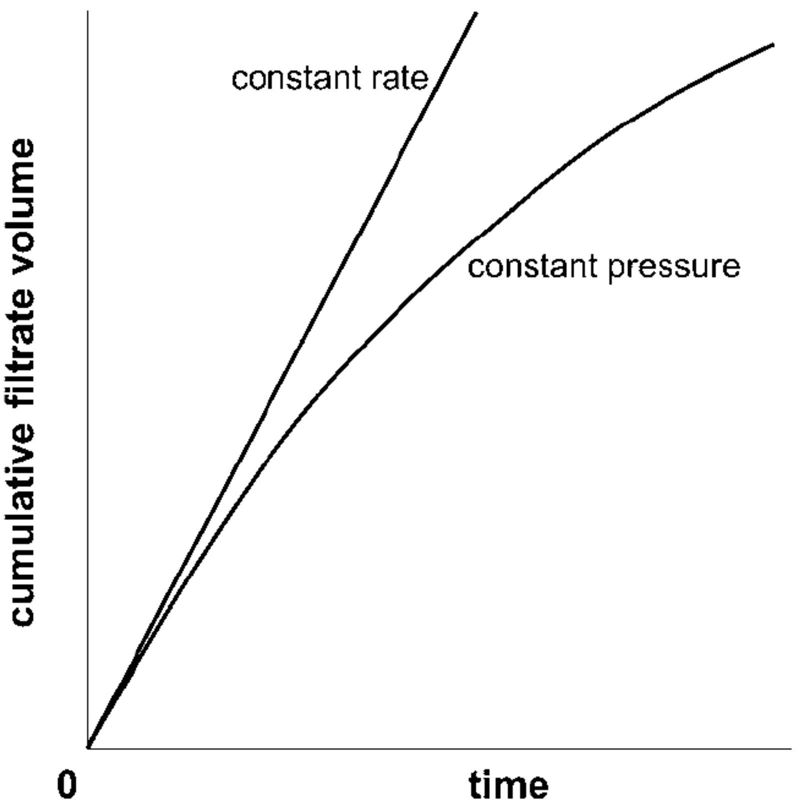
α - the **<u>specific cake resistance</u> [m/kg]**

<u>Darcy's Law with specific resistance:</u>

$$\eta \, \frac{R_C + R_M}{A} \, \frac{dV}{dt} \quad \Rightarrow \quad \Delta P = \frac{\eta\, c\, \alpha}{A^2}\, V \, \frac{dV}{dt} + \frac{\eta\, R_M}{A}\, \frac{dV}{dt}$$

Filtration – Incompr. Constant Pressure

Incompressible cake



Graph ***t/V* vs. *V*** to get:
the specific cake α and
filter medium resistance $\boldsymbol{R_M}$.

Constant pressure filtration,

After subsituting in Darcy's law and integration,

$$\int_0^t dt = \frac{\eta\, c\, \alpha}{A^2\, \Delta P} \int_0^V V\, dV + \frac{\eta\, R_M}{A\, \Delta P} \int_0^V dV$$

this yields the
Linearized parabolic rate law:

$$\frac{t}{V} = \frac{\eta\, c\, \alpha}{2\, A^2\, \Delta P}\, V + \frac{\eta\, R_M}{A\, \Delta P}$$

Filtration – Problem Solving 2

A slurry is filtered with a laboratory leaf filter with a filtering surface area of 0.05 m^2, using a vacuum pressure difference of 0.7 bar.
The slurry contains 5 vol% of solids (density 3000 kg/m^3)
Filtrate viscosity is 10^{-3} Pa.s (viscosity of water at room temp).
The volume of filtrate collected in the first 5 min was 250 cm^3 and, after a further 5 min, an additional 150 cm^3 was collected.
Determine the specific cake and cloth resistance.

A bit harder – but still …
Try to define the quantities for the equation yourself

The brain is a hard thing to change.
I'll guide you through this problem, do more yourself.

Filtration – Incompr. Constant Rate

Incompressible cake

$$\Delta P = \frac{\eta\, c\, \alpha}{A^2} V \frac{dV}{dt} + \frac{\eta\, R_M}{A} \frac{dV}{dt}$$

Constant rate filtration, subsituting in Darcy's law

$$\frac{dV}{dt} = \frac{V}{t} = \text{constant}$$

yields the

Pressure change with time:

$$\Delta P = \left(\frac{\eta\, c\, \alpha}{A^2} \frac{V}{t}\right) V + \left(\frac{\eta\, R_M}{A} \frac{V}{t}\right)$$

Characteristic Plot:

ΔP

vs **V.**

slope and intercept from such a graph provide the specific cake α and filter medium resistance R_M.

Liquid-Liquid Extraction and Solid-Liquid (Leaching)

Industrial liquid-liquid extraction systems

Solute	Carrier	Solvents
Acetic Acid	Water	Ethyl acetate, Isopropyl acetate
Aromatics	Paraffins	Diethylene glycol, Furfural, Sulpholane, NMP, DMSO
Caprolactam	Aqueous Ammonium Sulphate	Benzene, Toluene, Chloroform
Benzoic Acid	Water	Benzene
Formaldehyde	Water	Isopropyl ether
Phenol	Water	Benzene
Penicillin	Broth	Butyl acetate
Vanilla	Oxidized liquors	Toluene
Vitamins A, D	Fish liver oils	Liquid propane
Vitamin E	Vegetable oils	Liquid propane
Copper	Acidic leach liquors	Chelating agents in kerosene
Uranium	Acidic leach liquors	Tertiary amines in kerosene

What we will cover

By the end of this lecture you'll be able to:

Say what is **Drycleaning chemical P.** Other vocabulary: **gold panning, sluicing, Amalgamation, dissolution and recovery,**

Different generations of Drycleaning Equipment

Gold Extraction Processes
Mercury, Cyanide and the Cyanide recovery process

Coffee extraction processes
Turkish Coffee, Filter coffee, and
Espresso - also termperature, pressure, and crema!
Main process parameters in Espresso – **read articles** on best processing

<u>**Decaf Coffee??**</u>

Efficiency and cost comparisons – **find research articles** on process efficiency

Leaching Au out of ore

By the end of 1849 gold rush was at its full swing. Au was simply filtered from earth and rocks by mechanically sieving and picking. Obviously only larger particles of gold could be found this way.

Later Sedimentation in sluices was used – Smaller particles and poorer deposits explored

Leaching Au out of ore

Then came chemistry:

Au + Hg -> AuHg amalgam. Later Heat to get Au!

same reaction with Ag: Au and Ag often extracted together

Huge pollution in California with Hg from the Gold Rush

The cyanide process:

$4\,Au(s) + 8\,NaCN(aq) + O_2(g) + 2H_2O(l) \rightarrow$ (VERY TOXIC!)

$4\,Na[Au(CN)_2](aq) + 4\,NaOH(aq)$

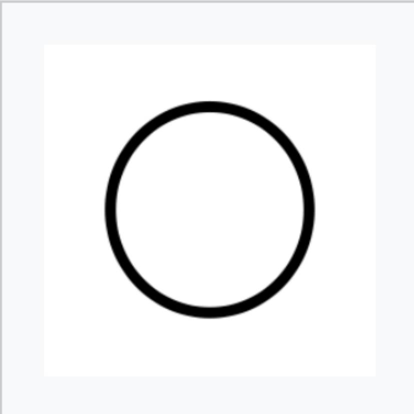
But can be treated with peroxomonosulphuric (Caro's) acid (H_2SO_5)

$CN^- + [O] \rightarrow OCN^-$ **(cyanide to cyanate)**

$OCN^- + 2\,H_2O \rightarrow HCO_3^- + NH_3$

Efficiency and cost comparisons – **we'll discuss research articles** on process efficiency

Drycleaning

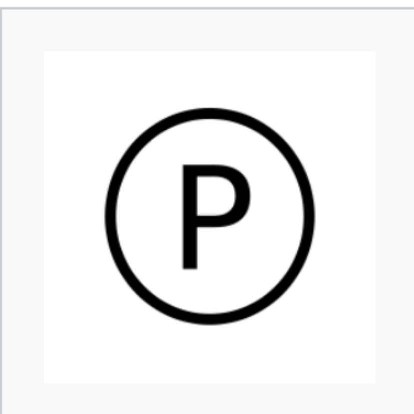


Professional cleaning symbol

Dry clean, hydrocarbon solvent only (HCS)

Gentle cleaning with hydrocarbon solvents

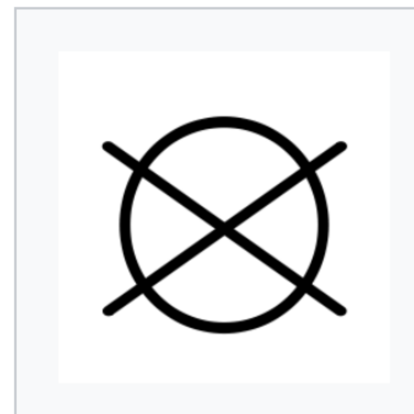
Very gentle cleaning with hydrocarbon solvents



Dryclean, tetrachloroethylene (PCE) only

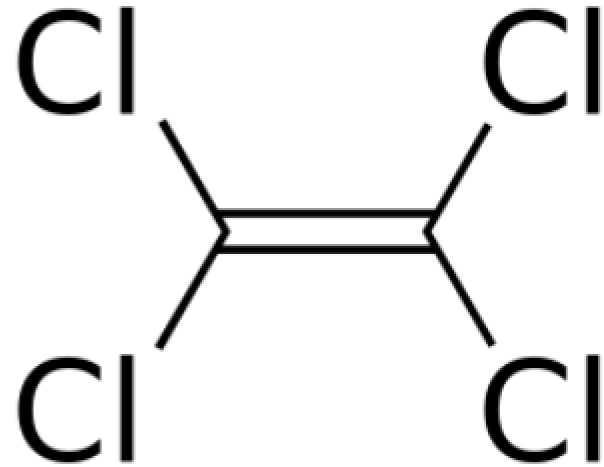
Gentle cleaning with PCE

Very gentle cleaning with PCE



Do not dry clean

Drycleaning



Process discovered in the 1930s. Tetra-chloro ethylene.

Non-Flammable. *Boiling-point*: 121 °C (O’Neil *et al.*, 2006)

P stands for per-chloro ethylene – all the H-s substituted by Cl

Strong bonds, don’t fall apart – non-flammable.

Coffee Extraction

7034 *J. Agric. Food Chem.* **2003,** *51,* 7034–7039

JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Chemical and Sensorial Characteristics of Espresso Coffee As Affected by Grinding and Torrefacto Roast

SUSANA ANDUEZA, M. PAZ DE PEÑA, AND CONCEPCIÓN CID*

Journal of the Science of Food and Agriculture

J Sci Food Agric **83**:240–248 (online: 2003)
DOI: 10.1002/jsfa.1304

Influence of extraction temperature on the final quality of espresso coffee†

Susana Andueza, Laura Maeztu, Lucía Pascual, Carmen Ibáñez, M Paz de Peña and Concepción Cid*

7426 *J. Agric. Food Chem.* **2002,** *50,* 7426–7431

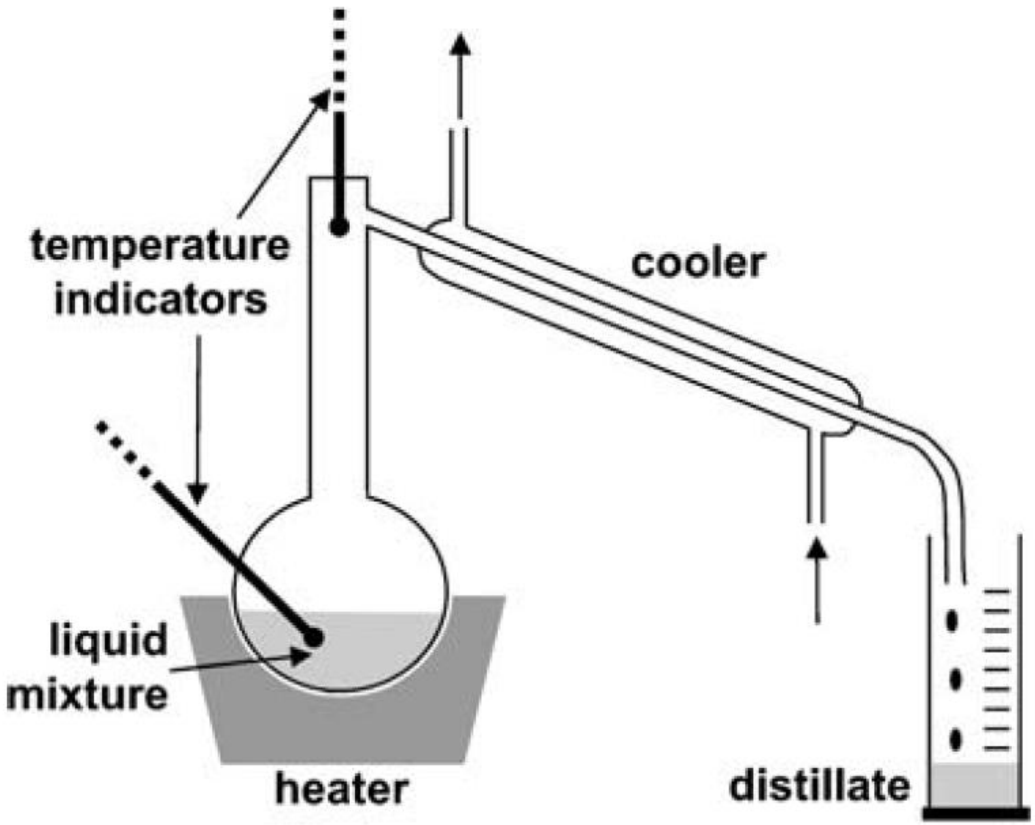
JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Influence of Water Pressure on the Final Quality of Arabica Espresso Coffee. Application of Multivariate Analysis

S. ANDUEZA, L. MAEZTU, B. DEAN, M. P. DE PEÑA, J. BELLO, AND C. CID*

Departamento de Bromatología, Tecnología de Alimentos y Toxicología, Facultad de Farmacia, Universidad de Navarra, E-31080 Pamplona, Spain

Distillation



From Benchtop to

Large Industrial Distillation Columns

What we will cover

By the end of this lecture you'll be able to:

Say what is **Distillation.** Other vocabulary: **flash distillation, multi-stage fractionation, vacuum distillation, vapor-liquid equilibria, Theoretical plates, liquid bubble point, vapor dew point, Reflux, rectification, boilup, distillation column, Rectification, Stripping sections, downcomer, reflux ratio, reboiler, Operating pressure**

McCabe-Thiele method for analyzing binary mixtures distillation

Multiple Component separation

Equipment building and operation

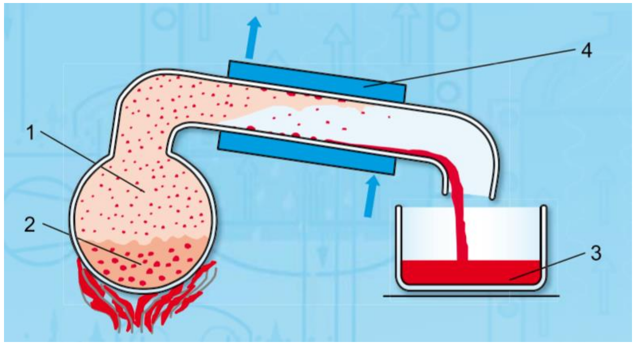
Process Efficiency and cost comparisons

Available on Knovel – in the library:
De Haan & Bosch, Industrial Separation Processes, 2013, de Gruyter (Berlin)
Distillation Fundamentals and Principles, Gorak & Sorensen, eds., 2014, Elsevier

Two-Phase Equilibria

General Separations

$$K_A \equiv \frac{x_{A,1}}{x_{A,2}}$$



In Distillation

$$K_i \equiv \frac{y_i}{x_i}$$

Equilibrium ratio of mole fraction of component A between phases 1 and 2

distribution coefficient Ki between phases x,y of component i

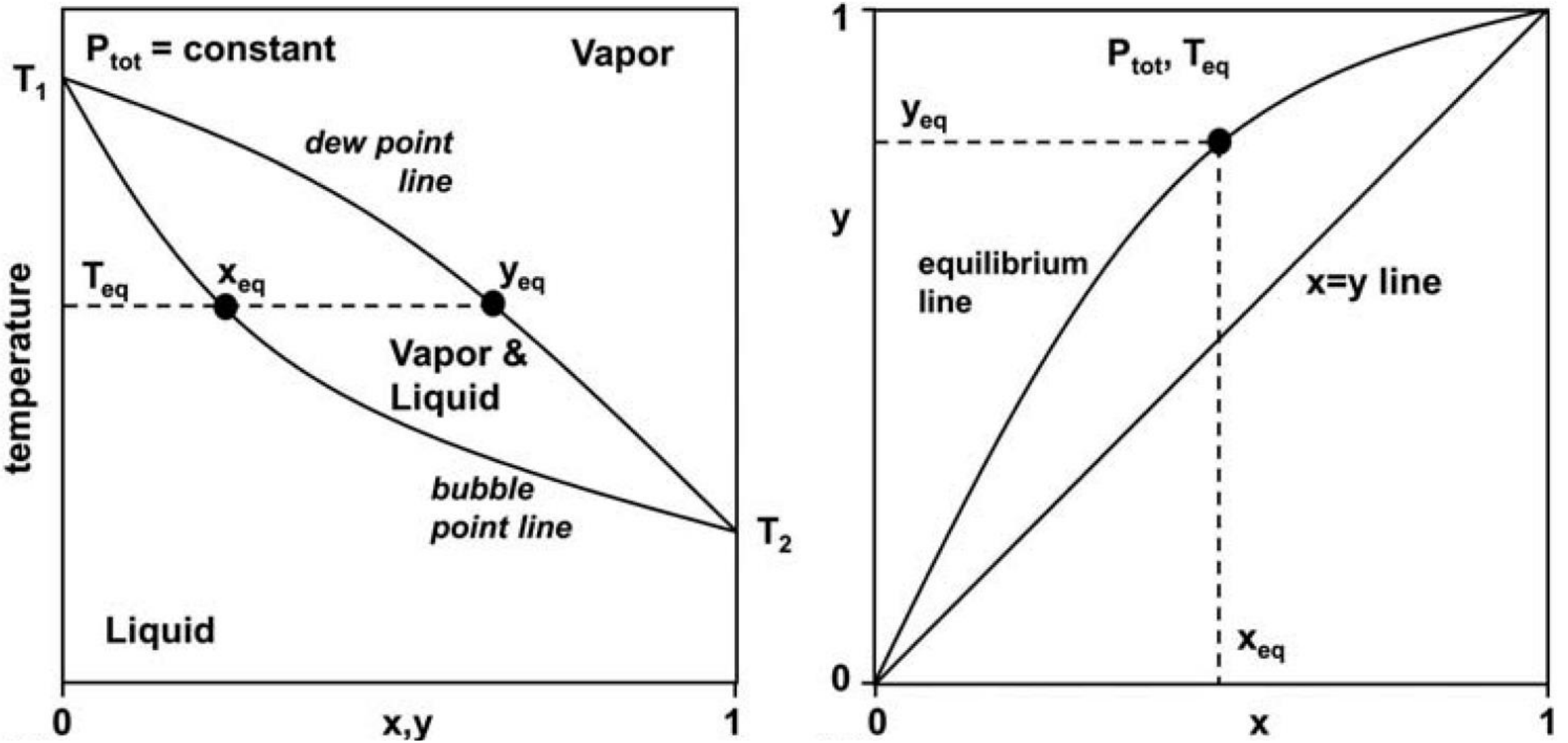
Separation factor and selectivity of separations of two components ***i and j***

$$\alpha_{ij} = \frac{K_i}{K_j} \; :$$

the **relative volatility** of ***i*** Determines the selectivity of Separation vs a reference ***j*** is

How do we measure relative volatility?

Two-Phase Equilibria



Constant pressure (isobaric) equilibrium diagrams and Changing compositions between phases

Two-Phase Equilibria

In Distillation

$$K_i \equiv \frac{y_i}{x_i}$$

$$\alpha_{ij} = \frac{K_i}{K_j}:$$

distribution coefficient Ki between phases x,y of component i

the **relative volatility** of ***i*** Determines the selectivity of Separation vs a reference ***j*** is

How do we measure relative volatility?

$$p_i = y_i P_{tot}$$

Dalton's law relates concentration of components in an ideal gas or to partial pressures in the vapor mixture

$$p_i = x_i P_i^0$$

Raoult's law gives partial pressure in the vapor (*pi*) from concentr. in the liquid (*xi*) mixture

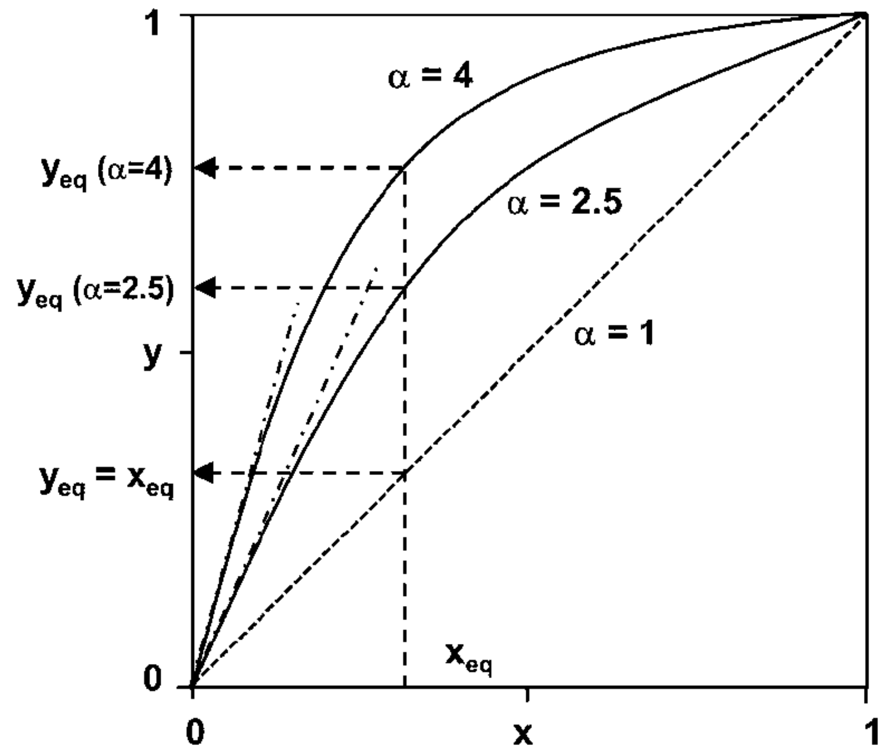
Combination gives:

$$y_i P_{tot} = x_i P_i^0 \qquad K_i = \frac{y_i}{x_i} = \frac{P_i^0}{P_{tot}} \quad \text{and} \quad \alpha_{ij} = \frac{K_i}{K_j} = \frac{P_i^0}{P_j^0}$$

Ideal vs Non-ideal Liquid Mixtures

Ideal liquid mixtures partial pressures in the vapor mixture

$$y = \frac{\alpha x}{1 + (\alpha - 1)\, x}$$



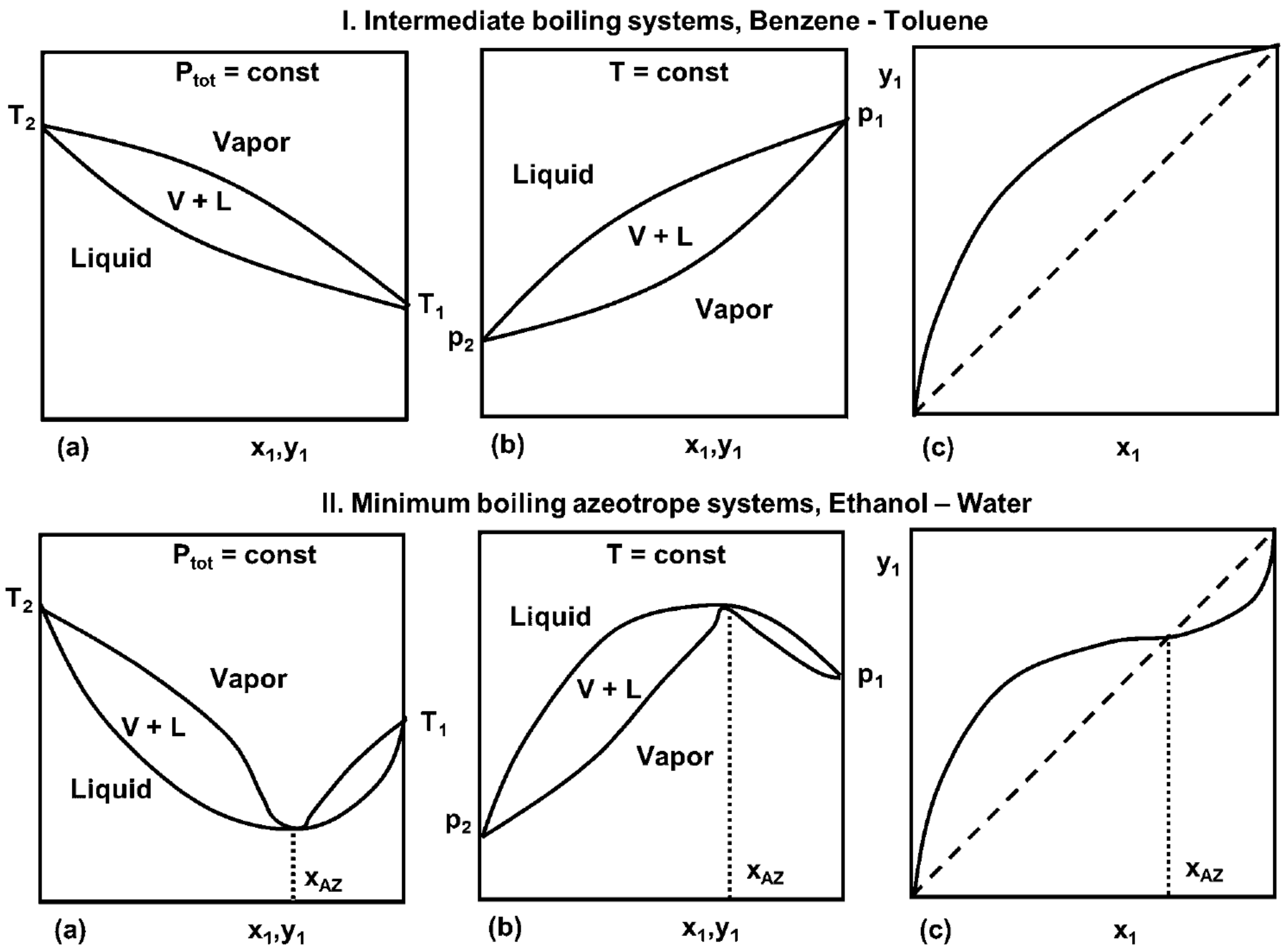
Non-ideal liquid mixtures
Require modified Raoult's law:

$$p_i = \gamma_i x_i P_i^0$$

Where γ_i is a correction factor, the **liquid phase activity coefficient**

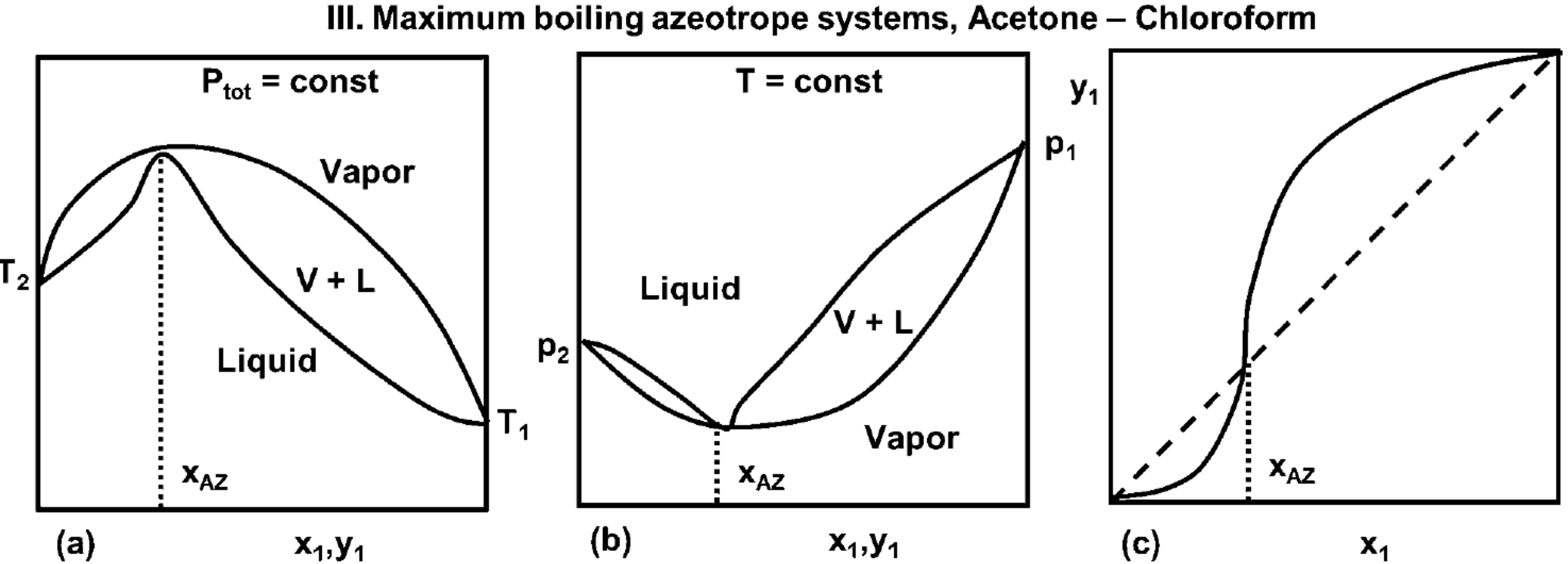
What happens when γ_i Becomes very high?? Potential for **azeotropes** and separation problems.

Azeotropes and Problem Separations



Ideal vs azeotropic mixtures

Azeotropes and Problem Separations



Simple distillation is not effective for separating **azeotropic mixtures** (mix T_b not between components)
We'll cover techniques for their separations later but in brief need adsorption, extraction, etc., i.e.:
Need to combine distillation with other separations

Simple vs. Flash Distillation

Same, yet

Batch vs. Continuous

$$y = \frac{\alpha x}{1 + (\alpha - 1) x} \quad \text{with} \quad \alpha = \frac{K_A}{K_B}$$

Single stage separation efficiency.
Relative volatility *α* is the equilibr.
VL- constant, also referred to as *K*

continuous feed is partially vaporized to give a vapor richer in the more volatile components.
limited degree of separation
Uses:e.g. seawater desalination,
Where bp differences are high

Above is derived from Two Mass Balances –
One for whole liquid, one for a component in the liquid

$$F = V + L \quad \text{and} \quad Fz = Vy + Lx$$

Here F is the total fluid and
V, L – vapor and liquid separated

z, y, x, and the mole fractions of
a component in fluid, vapor, liquid

Single vs. Multi-Stage

Rearranging this equation yields form on the right

$$y = \frac{\alpha x}{1 + (\alpha - 1)\,x} \quad \text{with} \quad \alpha = \frac{K_A}{K_B}$$

$$\frac{y_0^*}{1-y_0^*} = \alpha \cdot \frac{x_0}{1-x_0}$$

What if we repeat this again?

Subscript 0 refers to initial state

New liquid has $x_1 = y_0$, so compare composition of 1st and 2nd liquids:

$$\frac{x_1}{1-x_1} = \alpha \cdot \frac{x_0}{1-x_0}$$

Similarly if we repeat a second time Composition gets even purer:

$$\frac{x_2}{1-x_2} = \alpha \cdot \frac{x_1}{1-x_1} = \alpha^2 \cdot \frac{x_0}{1-x_0}$$

For the nth step:

$$\frac{x_n}{1-x_n} = \alpha^n \frac{x_0}{1-x_0}$$

or rearranging:

$$x_n = \frac{\alpha^n \cdot x_0}{1 + (\alpha^n - 1) \cdot x_0}$$

Even small α can lead to pure product!

Stichlmair, J. (2000) Distillation&Rectification, Encyclopedia of Industrial Chemistry, DOI: 10.1002/14356007.b03_04

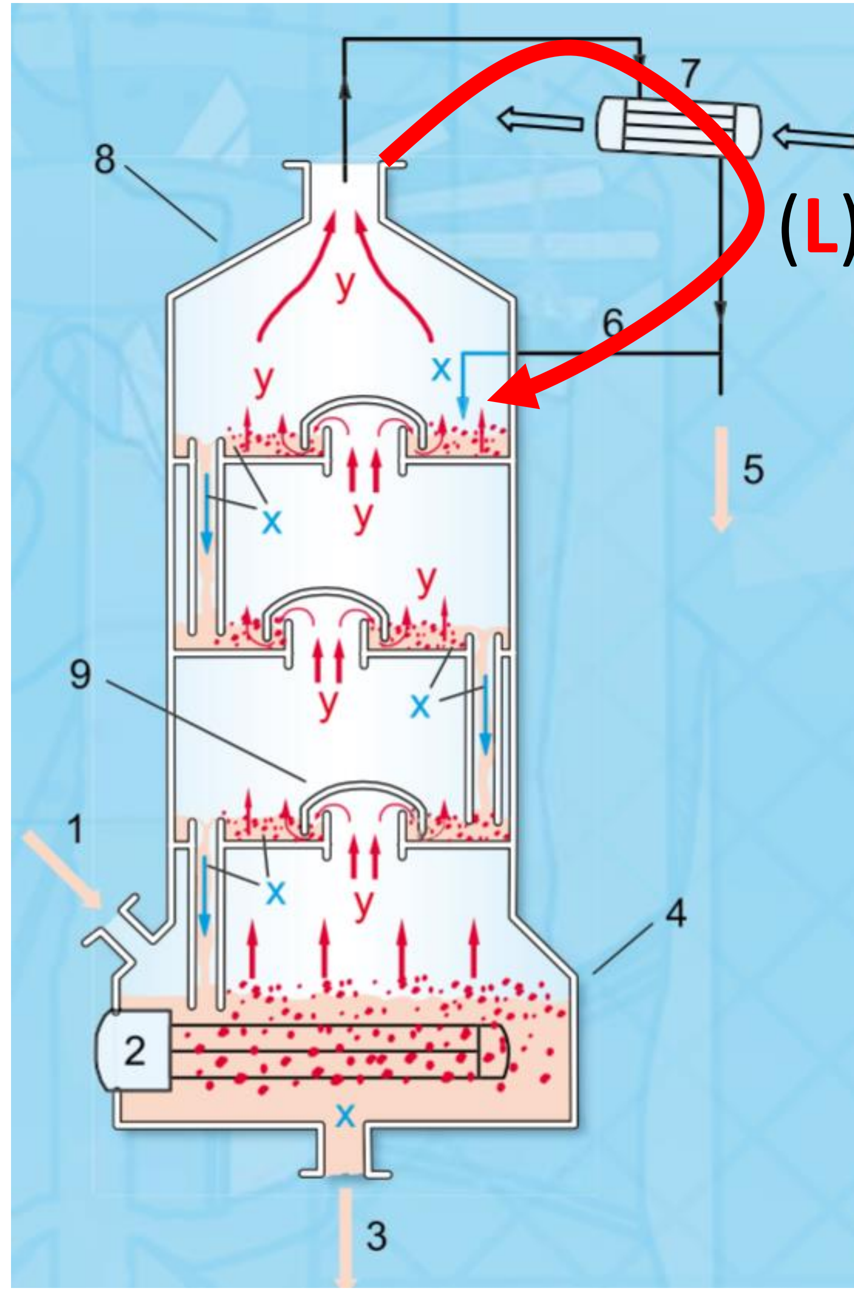
Multi-Stage and Column Distillation

with Rectification

Top vapor = product

Part condensed, fed back

Words for vocabulary:
distillation column, Reflux, rectification, reflux ratio, reboiler, Operating pressure



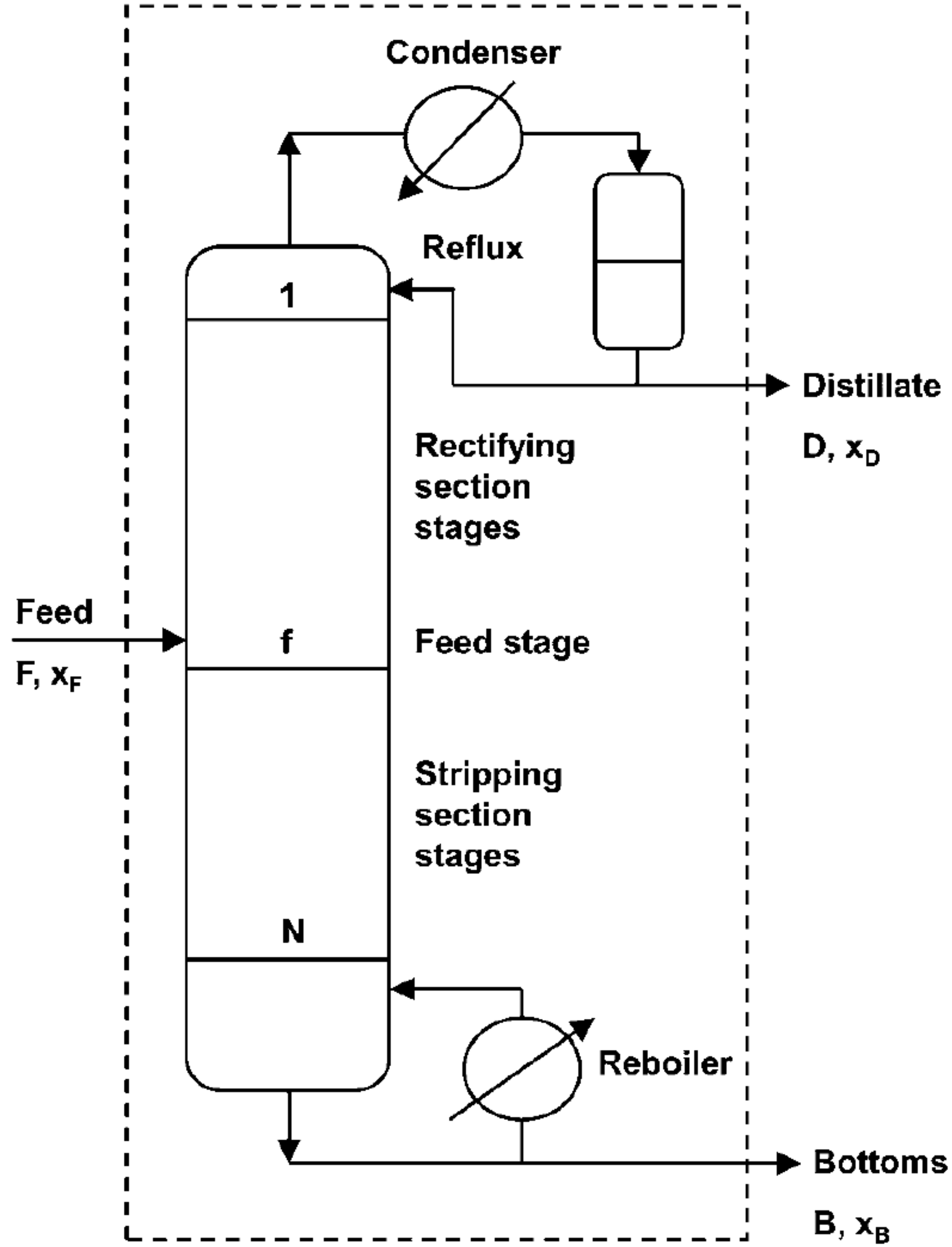
The **Reflux ratio** **R = L/D** is the ratio of reflux flow (**L**) to distillate flow (**D**)

(**L** Being returned back to the column for re-distilling)

Multi-Stage and Column Distillation

Rather than only going up
One can also come down
From the feed
Concentrate the less volatile
Components.

Keywords – downcomer,
Stripping sections
Rectifying sections



Operating condition: T and L/V
Graphical Determination – Mole Fractions

When we specify T, we can also
Decide on q

$$q = \frac{L}{F}$$ = Fraction of feed remaining liquid

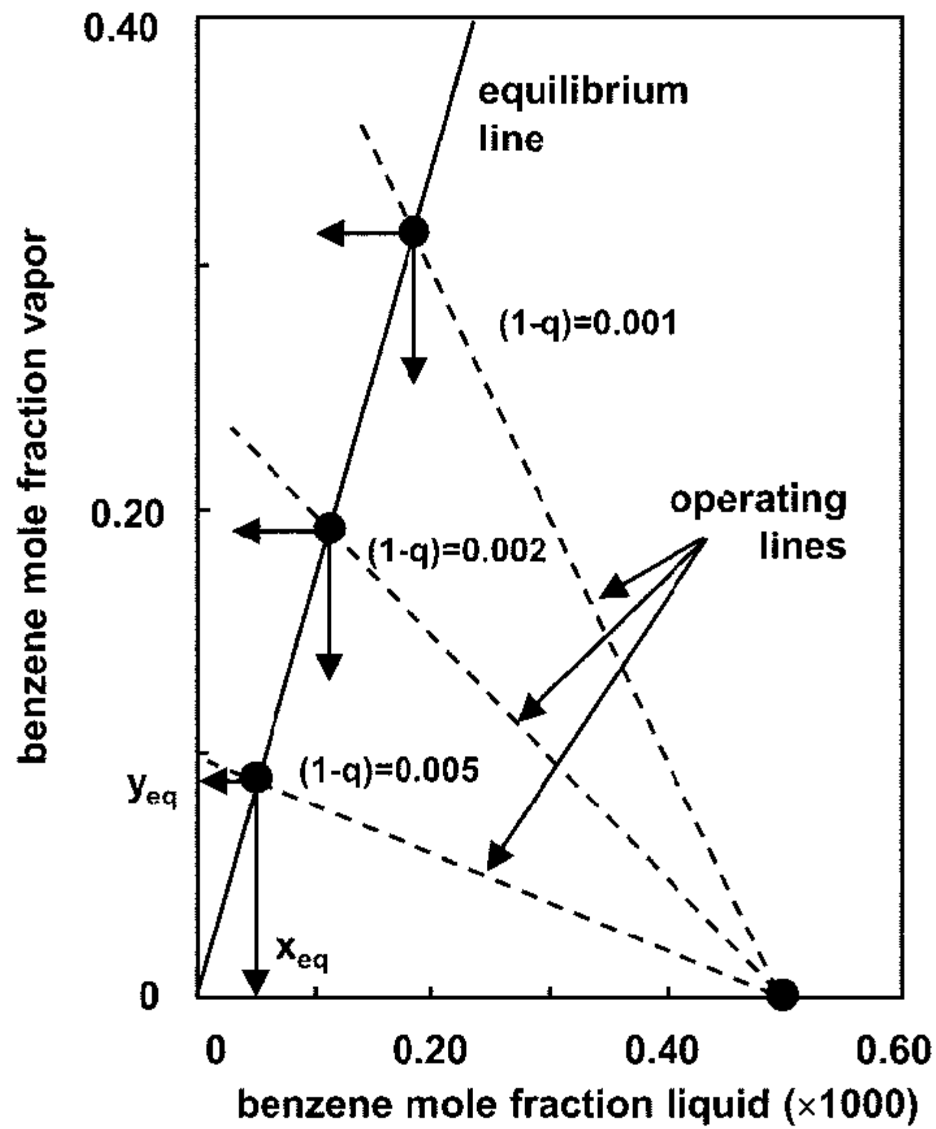
(1-q) = V / F (Fraction vaporized)

The **Operating Line equation** is:

$$y = -\frac{q}{1-q}x + \frac{1}{1-q}z$$

The **Equilibrium line** is:

$$\frac{y}{1-y} = \alpha \cdot \frac{x}{1-x}$$



Operating condition: T and total Pressure P
Graphical Determination – Mole Fractions

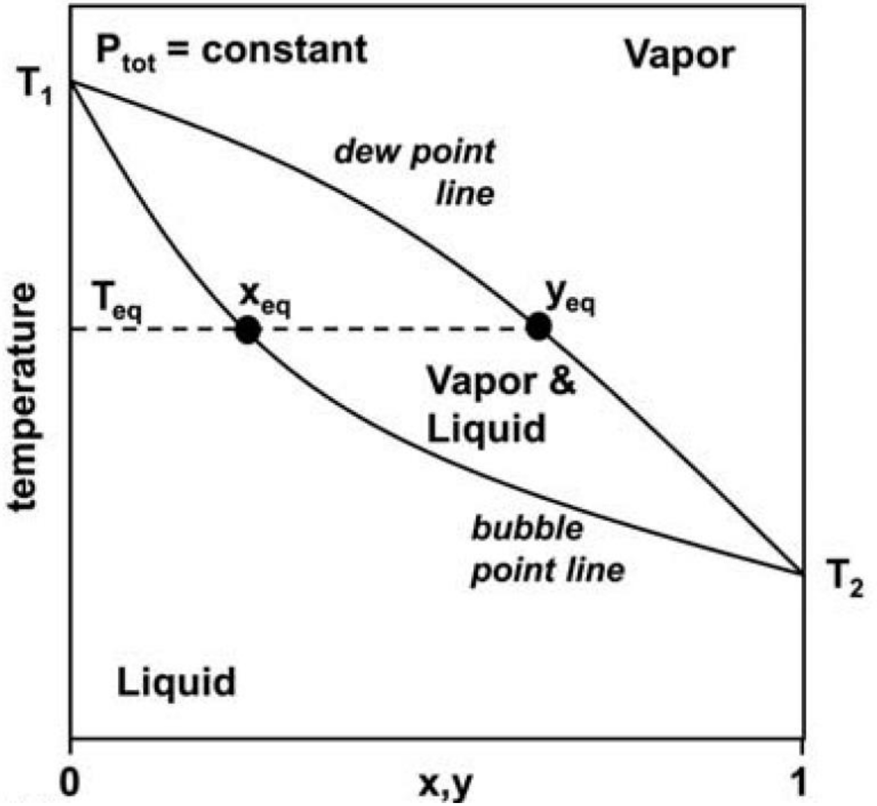
Now we have to find q

$q = \frac{L}{F}$ = Fraction of feed remaining liquid

by eliminating x, y from the

Operating Line equation:

$$y = -\frac{q}{1-q}x + \frac{1}{1-q}z$$



And **Equilibrium line**:

$$\frac{y}{1-y} = \alpha \cdot \frac{x}{1-x}$$

given T , P_{tot} – the K_i are known constants, so

$$q = -z\frac{K_B}{1-K_B} - (1-z)\frac{K_A}{1-K_A} \quad \text{and} \quad x = \frac{1-K_B}{K_A-K_B}$$

McCabe-Thiele Analysis

Internal balances for the Rectifying section

In vapor, liquid and distillate $V'_{n+1} y_{n+1} = L'_n x_n + D x_D$

Operating line is:

$$y_{n+1} = \left(\frac{L'}{V'}\right) x_n + \left(\frac{D}{V'}\right) x_D$$

or

And since

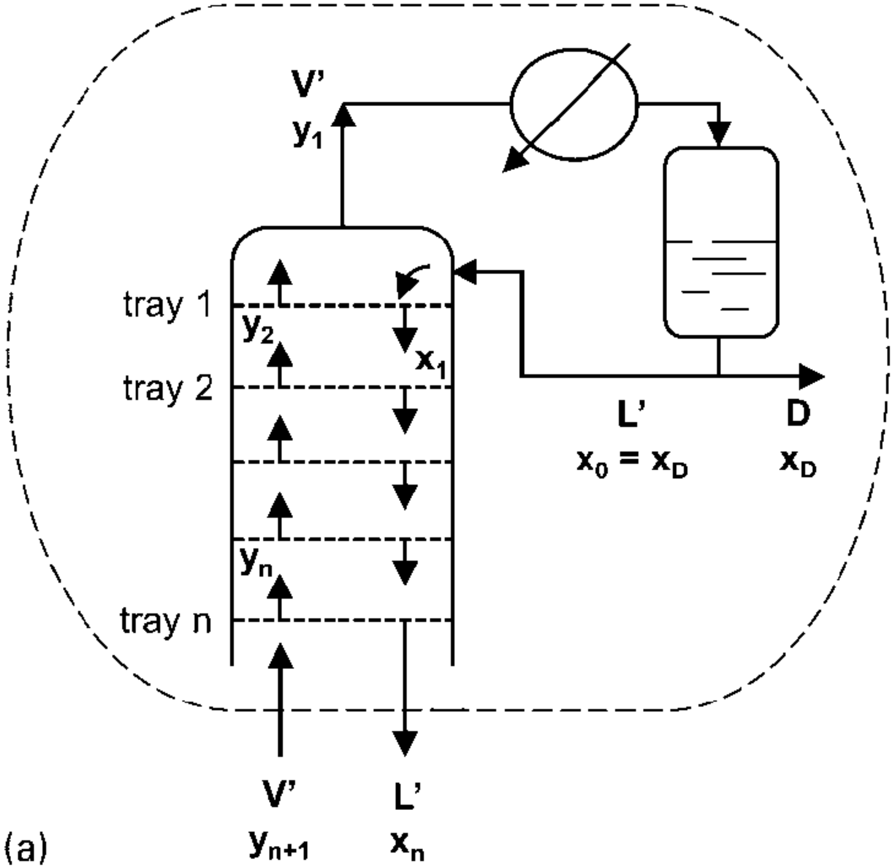
$V' = L' + D$ and

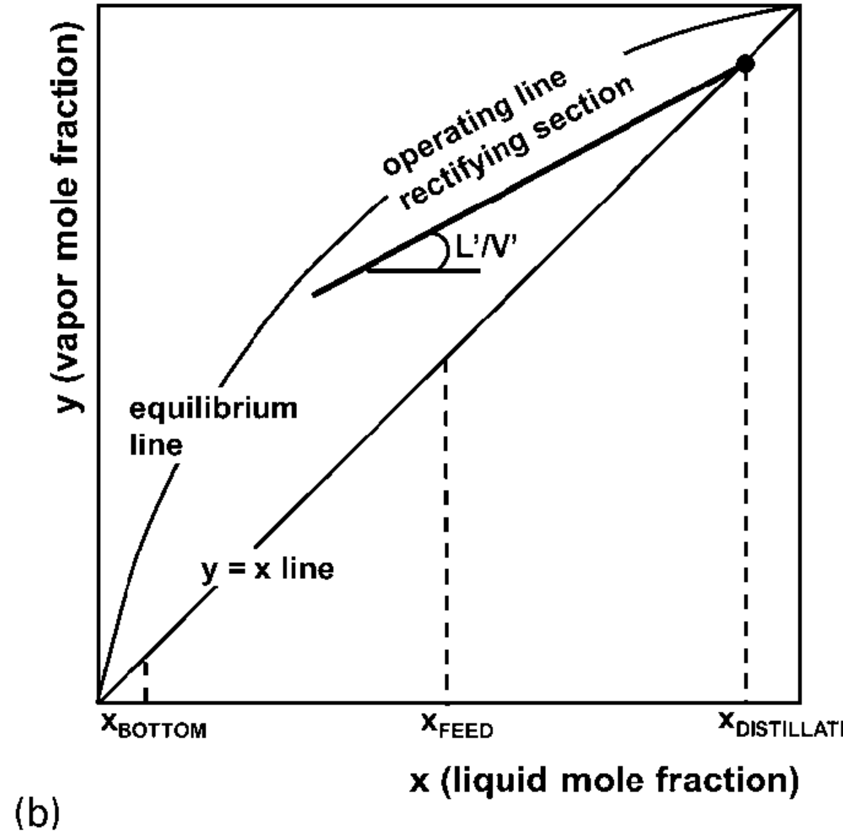
$L'/D = R$

$$y_{n+1} = \left(\frac{R}{R+1}\right) x_n + \left(\frac{1}{R+1}\right) x_D$$

This information is much easier to view graphically as we'll see next.

McCabe-Thiele Analysis





the **operating line** is a straight line in the *y*-*x*-diagram with an intersection at $y = x_D$ on the $y = x$ line, for **specified values of *R* and x_D** (purity of distillate)

McCabe-Thiele Analysis

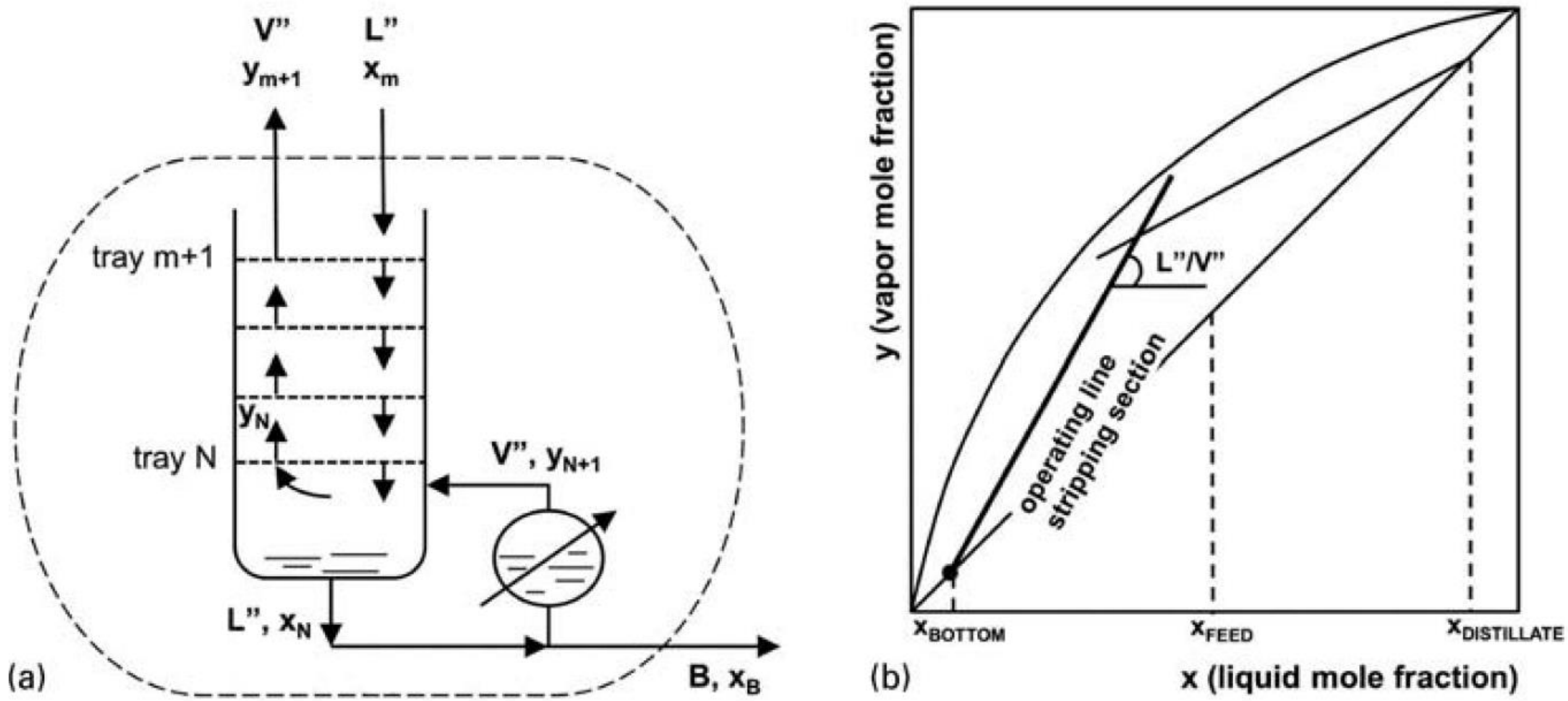
Internal balances for the Stripping section

In vapor, liquid and bottoms $L''_m x_m = V''_{m+1} y_{m+1} + B x_B$

Operating line is:

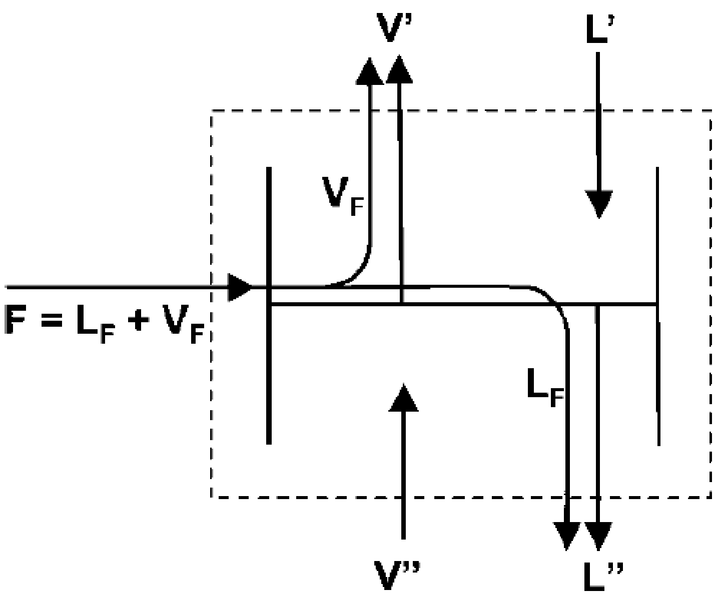
And graphically:

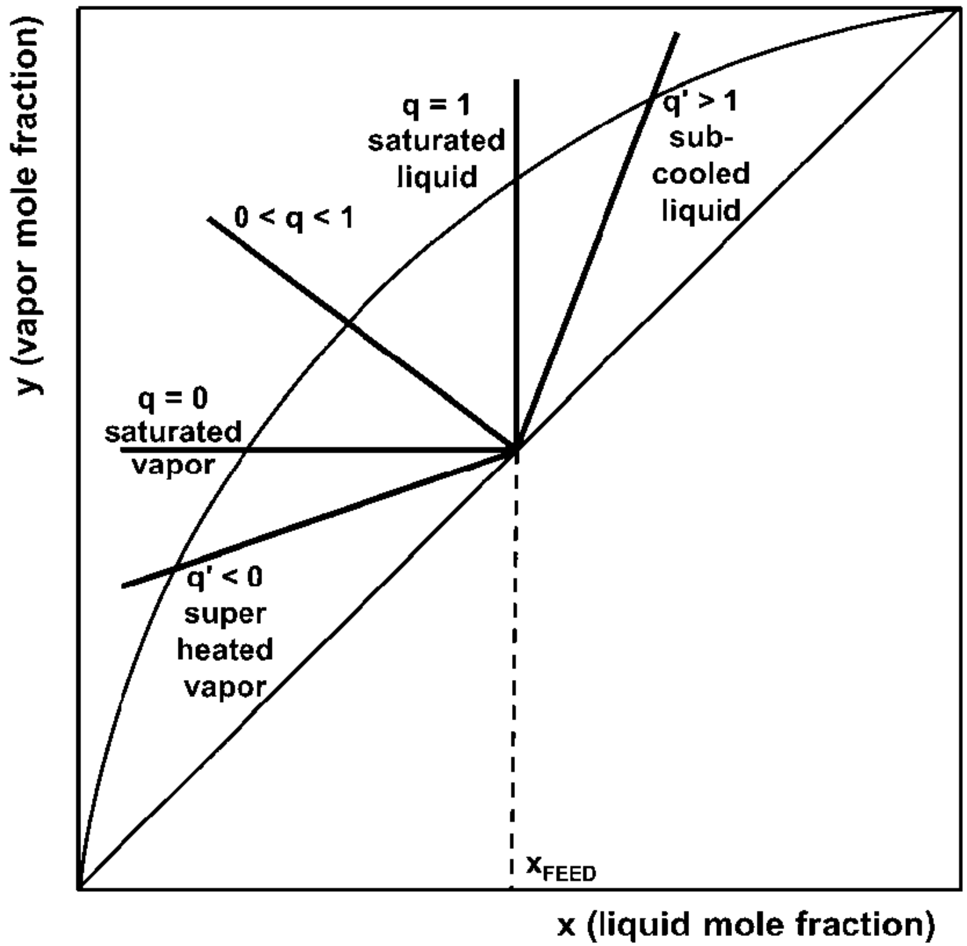
$$y_{m+1} = \left(\frac{L''}{V''}\right) x_m - \left(\frac{B}{V''}\right) x_B$$



McCabe-Thiele Analysis

Dependence of q-line on the feed condition:



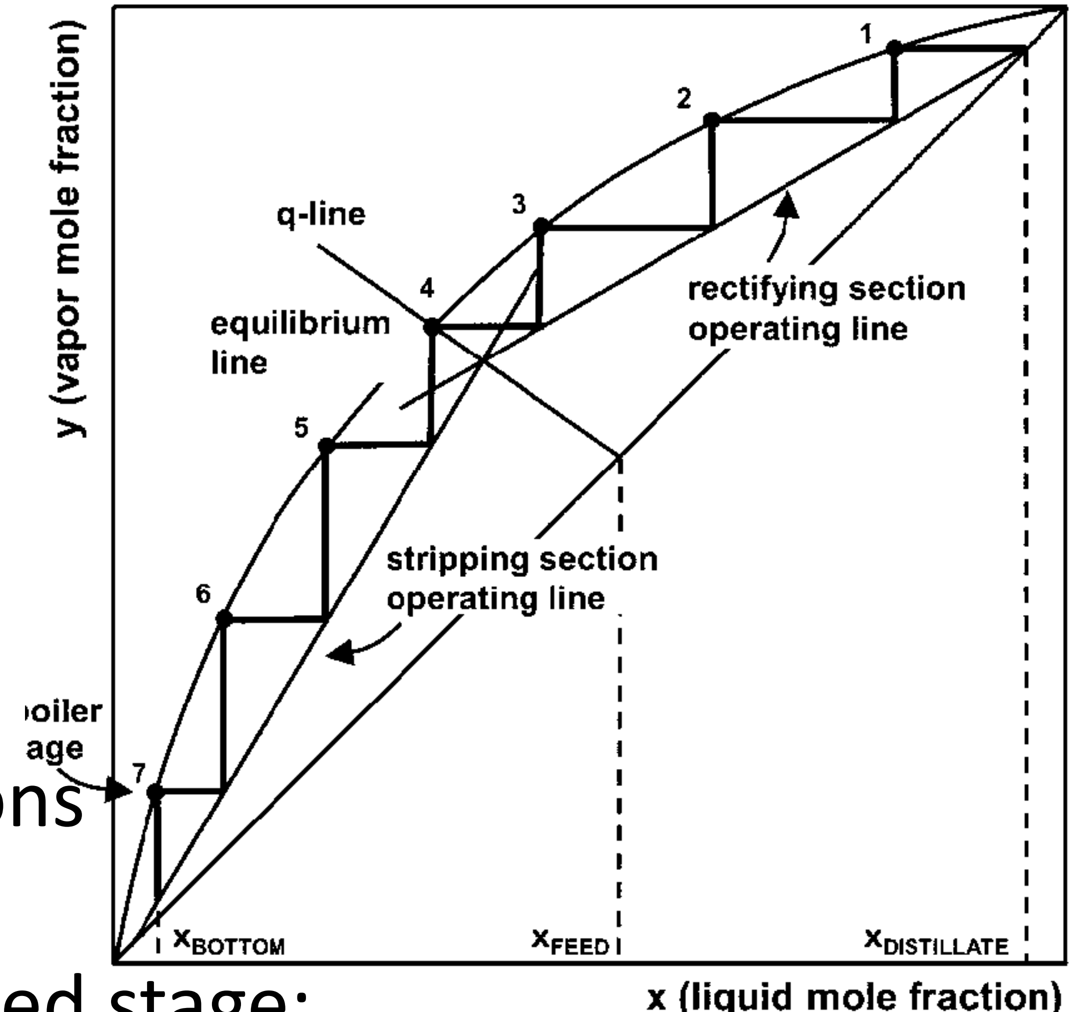


McCabe-Thiele Analysis

Graphical Determination of # of equilibrium stages:

First:
Construct a staircase
Between
Operating lines and
Equilibrium curve.

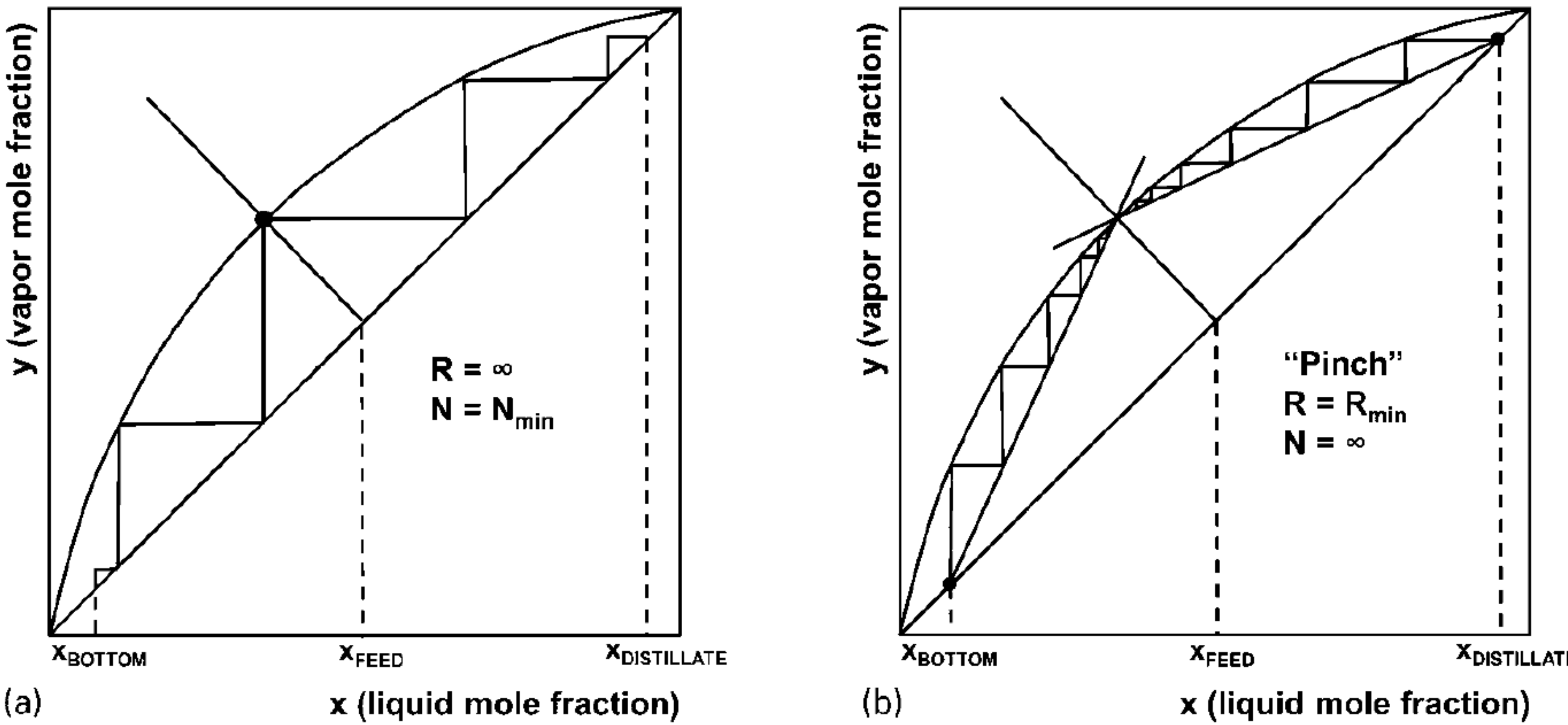
Horizontals – equil.
Verticals – compositions
Passing each other



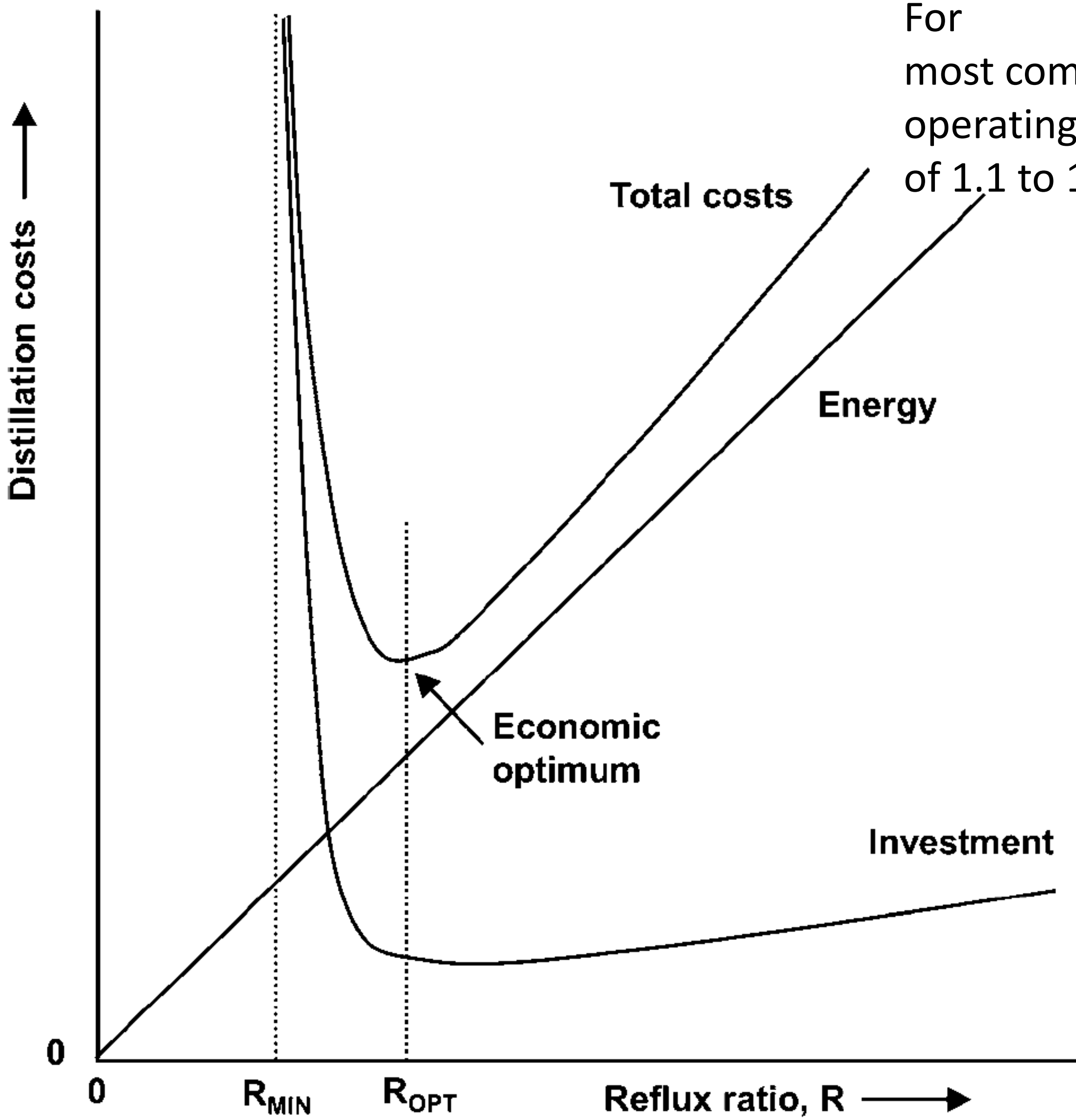
Optimal location of feed stage:
First location after horizontal crosses the q-line.

McCabe-Thiele Analysis

Minimum # stages vs. Minimum reflux ratio



McCabe-Thiele Analysis



For
most commercial operations the optimal
operating reflux ratios are in the range
of 1.1 to 1.5 times the minimum reflux ra

Operational cost

McCabe-Thiele Analysis

$$R_{\min} = \left(\frac{L'}{D}\right)_{\min} = \left(\frac{L'}{V' - L'}\right)_{\min} = \frac{\left(\frac{L'}{V'}\right)_{\min}}{1 - \left(\frac{L'}{V'}\right)_{\min}}$$

For most commercial operations the optimal operating reflux ratios are in the range of 1.1 to 1.5 times the minimum reflux ratio.

Operational cost

Distillation Problems

Practice

Remember – Two tutorials next Thursday